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Enhancing Adhesive Bonding and Mechanical Properties of Composite Sandwich Panels through Atmospheric Plasma Activation

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Abstract

This study investigates the effect of Atmospheric Plasma Activation (APA) treatment on the thermomechanical performance of adhesively bonded skins of composite sandwich panels. The investigations show that APA treatment enhances surface wettability through the water contact angle measurements, and adhesion between the skin and honeycomb core of the panels under mechanical tests. Specifically, APA treatment results in an 11% increase in flatwise tensile strength and a 12% enhancement in impact strength of the sandwich laminates. Flatwise tensile tests reveal superior tensile strength and toughness in APA-treated specimens, with fracture patterns suggesting more robust adhesion. Charpy impact test results confirm increased energy absorption, reflecting better mechanical resilience. Scanning Electron Microscopy (SEM) and Dynamic Mechanical Analysis (DMA) techniques are utilized to validate these findings. Overall, APA treatment proves to be an effective technique for enhancing the performance of composite sandwich panels, offering improved adhesion, strength, and impact resistance. This approach holds significant promise for advancing high-performance composite materials in various engineering applications.

Keywords: Composite Sandwich Panels, Atmospheric Plasma Activation (APA), Flatwise Tensile Test, Adhesive Bonding

Highlights

- APA treatment enhances wettability and adhesion in sandwich panels.
- Improved adhesive distribution between skin-core after sandwich manufacturing.
- 11% increase in tensile strength and 12% in impact strength with APA
- Better toughness and energy absorption via SEM and DMA support in APA.

1. Introduction

Honeycomb sandwich structures are composite configurations that consist of two relatively thin skins bonded to a comparatively thick lightweight honeycomb core. The skin layers predominantly endure tensile and compressive forces, while the core layers effectively withstand buckling and provide resistance against out-of-plane shear loads [1,2]. Sandwich structures offer many advantages, including commendable corrosion resistance [3], endurance to thermal conditions, remarkable energy absorption capabilities [4], a high stiffness-to-weight ratio and robust buckling resistance [5].

The selection of suitable materials for the core and skin components in sandwich structures is a critical parameter which impacts the holistic stiffness and weight characteristics of the manufactured parts. A review of numerous scientific studies reveal that many types of materials are utilized as skin layers and core materials in the sandwich structures [6]. A thorough analysis of the materials employed in the aviation industry shows that glass fiber (GFRP) and carbon fiber reinforced polymer (CFRP), also Kevlar reinforced laminates are extensively employed as skin materials. Upon considering the diversity of core components used in sandwich structures, it is observed that Nomex honeycomb, aluminum honeycomb and foam core materials are commonly manufactured in the aerospace applications [7].

Nomex paper strip honeycomb cores are frequently employed in sandwich structures owing to their outstanding lightweight characteristics and notably elevated level of the mechanical performance [8,9]. Nomex honeycomb core sandwich structures have wide-ranging applicability as secondary components in various sections of aircraft, such as floors, doors, aerodynamic fairings, overhead storage bins, ceiling or sidewall panels, control surfaces (rudders, ailerons), spoilers, nacelles and other related components. [2,10–12].

Sandwich structures can be assembled through the utilization of secondary bonding process or a co-curing process [13]. The manufacturing of honeycomb core sandwich structures using the secondary bonding method involves a two-step process. Initially, the prepreg skins are individually cured to attain the desired structural properties. Subsequently, these cured skins are bonded to the core material by applying an adhesive film, ensuring the formation of a robust and cohesive sandwich structure. The co-curing process achieves consolidation and bonding simultaneously in a single step [14]. Despite its advantages as a one-step manufacturing process, co-curing introduces complexity due to the interplay of physical phenomena such as skin consolidation, film adhesive flow, and gas pressure within the core. These interactions give rise to complications such as resin infiltration into the core, adhesive ingress into the prepreg and the potential formation of voids in the cured adhesive [15]. This leads to a reduction in the mechanical performance, especially compression stiffness and strength of the laminate.

The process of bonding the skins with the honeycomb core in sandwich composites holds paramount significance in determining the overall performance of the structure. To achieve this, fasteners and adhesives, such as paste and film varieties, are utilized as means of effectively joining these two constituent components [16]. The adhesive serves the purpose of affixing the skins to the honeycomb core, enabling the efficient transfer of stress from one skin to another by means of the honeycomb core [17]. Skin-core debonding is a critical occurrence for the integrity of sandwich structures, as it hinders the proper transmission and distribution of external loads, thereby impacting the load-bearing capacity and balance of the system [18]. Moreover, uniform application of adhesive enhances the load-carrying capacity of the sandwich structure by supporting the above-mentioned load transfer mechanism.

Another technique, atmospheric plasma activation (APA) has proven to hold potential compared to the traditional surface preparation methods that leads to high-strength and durable adhesively-bonded joints [19–21]. APA is a highly effective, economically viable, and environmentally friendly technique that improves mechanical performance of the bonded joints by increasing the surface free energy (SFE) and wettability of adherend surfaces without causing detrimental effects or damage to the materials [22–24]. The application of APA results in the partial decomposition of the hydrophobic layer on the adherend surface, leading to the formation of new hydrophilic groups which favor the chemical bonds to be formed with the adhesive layer. This transformation is reported to be a vital aspect to increase the surface energy, among others; and is generally observed in the reduced levels of water contact angle values [25].

In the literature, there are several studies focusing on the mechanical performance evaluation for different types of sandwich composite structures. Zaharia et al. [26] undertook a comprehensive investigation of CFRP skin - Nomex sandwich composites in their study, encompassing a range of mechanical tests such as Charpy impact testing, flatwise compression testing, and fatigue testing. The thickness of Nomex core was 5 mm, while the overall sandwich structure had a total thickness of 9 mm. By subjecting the CFRP-Nomex sandwich structure to the Charpy impact test, an average impact strength of 54.4 kJ/m^2 was determined for the CFRP-Nomex sandwich specimens. It was observed that the specimens had a maximum achievable impact strength value of approximately 70 kJ/m^2. Here the CFRP skins are stated to be bonded with no surface pretreatment. Muniraj et al. [27] conducted the determination of specific energy absorption values utilizing the Charpy impact test methodology, as well as the evaluation of residual compressive strength values and damage assessment of honeycomb composite sandwiches. The honeycomb sandwich structure consisted of an aluminum AA 3003 core material and a glass fiber composite skin. Various configurations of honeycombs were employed to investigate their effect on the overall mechanical performance of the structure, including different core heights, cell sizes, cell thicknesses, and E glass fiber composites with both random and predetermined orientations. The results showed that densely packed thicker honeycombs exhibited greater fracture energy. Similarly, Liu and Gao et al. [28] investigated the relationship between skin thickness, honeycomb core orientation, and the collapse process of CFRP skin - Nomex core composite sandwich structures using experimental methods. When comparing sandwich structures with identical skin thickness but different core orientations, the energy absorption capability was found to be superior in the sandwich structure with an L-oriented core compared to the sandwich structure with a W-oriented honeycomb core. Furthermore, for sandwich structures with the same core orientation, the energy absorption of the thick-shell sandwich structure was observed to be substantially greater than that of the thin-shell sandwich structure. In another study, Liu et al. [29] performed experimental study for Nomex honeycomb sandwich composites under transverse loading. The experimental setup involved the use of a sandwich structure comprising a 20 mm Nomex core and a 1.88 mm carbon fiber epoxy laminate for conducting tensile flatwise tensile test. The average tensile strength recorded during the tensile tests was 2.13 MPa. In the compression flatwise tests, a Nomex honeycomb with thicknesses of 20 mm and 14 mm, both with the same skin thickness, was utilized. The compressive loads of honeycomb cores with varying core heights are nearly identical, both around 4300 N, indicating compressive strengths of approximately 1.72 MPa. Moreover, Widagdo et al. [30] performed an investigation to assess the flatwise tensile properties of sandwich composite structures comprised of fiberglass honeycomb cores and carbon fiber epoxy skins. The honeycomb cores were prepared using various parameters for core material preparation. the honeycomb sandwich structures gave an average flatwise tensile strength of 5.59 MPa. Zhu et al. [31] conducted a study involving the fabrication of sandwich composite structures utilizing a Nomex core. Two types of skin were employed: a hybrid face layer consisting of a quartz fiber reinforced plastic (QFRP) laminate and two copper layers, and a QFRP laminate without copper layers. The mechanical performance of the composite sandwich panels was evaluated in four aspects: the flatwise tensile strength test, the edgewise compressive strength test, the three-point bending test and the tensile plate shear test. The experimental data revealed that the presence of copper layers in the sandwich panel resulted in lower values for all four mechanical properties, varying in proportions. The flatwise tensile strength showed the largest reduction, with a decrease of 26.1%. However, there is a noticeable gap in the literature regarding efforts to enhance the structural integrity of sandwich composites by improving the bonding performance between the core and skin.

Since the processing parameters of the skin and core may not always be compatible, the co-curing method cannot always be utilized. Therefore, there is a requirement in the industry to implement a secondary bonding process for the manufacturing of sandwich composites. As previously indicated, the optimization of bonding process is one of the most crucial factors affecting the mechanical properties of sandwich structures. Therefore, in this study, the effect of APA treatment on the bonding performance between the core and shell and thus on the mechanical properties of secondary bonded sandwich composites is investigated for the first time in literature. Sandwich composite specimens are prepared with APA-treated and non-treated CFRP skins bonded on Nomex honeycomb core, followed by a series of characterization and mechanical tests, namely, dynamic mechanical analysis (DMA), flatwise tensile testing, and Charpy impact testing. This comprehensive investigation is further supported by microscopic analysis to observe the effects of APA treatment in the produced specimens by means of adhesive morphology and the corresponding failure mechanisms in the damaged specimens. The results demonstrate a significant improvement in the structural integrity of the composites, highlighting the effectiveness of APA treatment in enhancing bonding performance.

2 Materials and Methods

2.1 Materials

For this study, the core material, AHN-4120-1/8-3.0 aramid fiber/phenolic coated Nomex honeycomb [32], and the skin material, 6.35 mm wide AX-201XL epoxy carbon slit-tapes [33], are acquired from KORDSA. The slit-tapes consists of Mitsubishi 34700 12K carbon fiber bundles and a hotmelt epoxy resin system resulting in an overall resin weight content of 35%. The adhesive film used for bonding the CFRP skins to the Nomex honeycomb core is Scotch-Weld AF 163-2 K, manufactured by 3M Company [34]. Technical grade Isopropyl alcohol (IPA) and Solid sodium hydroxide (NaOH) with a density of 2.13 g/cm^3, as well as an 85 wt.% phosphoric acid solution obtained from Sigma-Aldrich, are utilized for cleaning the aluminum blocks used during the flatwise test. Furthermore, to bond the aluminum alloy blocks of the flatwise tensile test specimens, Scotch-Weld DP 490 epoxy structural adhesive from 3M Company, is employed.

2.2 Methods

2.2.1 Carbon Fiber Epoxy Composite Manufacturing

The composite laminates forming the skin of the sandwich structures are manufactured using automated fiber placement (AFP) for the lay-up process, followed by oven curing. First, slit tapes with a unidirectional (UD- [0]8) stacking sequence are placed using a Coriolis C1 (Coriolis Composites) AFP system with a KUKA QUANTEC KR210 R3100 robot (Figure 1a). For the AFP lay-up process, a low consolidation force of 300 N and a feeding rate of 0.1 m/s are employed [35]. The uncured UD composite laminate is placed in a vacuum bag for 24 hours at 1 atm pressure for debulking before oven curing. After debulking, the oven curing process is applied to the laminates using the curing cycle recommended by the manufacturer (Figure 1b)

[33]. The curing process consists of two dwell periods at 70°C and 120°C. First, the temperature is gradually increased to 70°C with a rate of 1°C/min and the laminates are kept at that temperature for 30 minutes. Following this, the temperature is further increased to 120°C at the same rate, with the laminates being held for 45 minutes. Lastly, the laminates are cooled to 50°C at a rate of 1°C per minute.

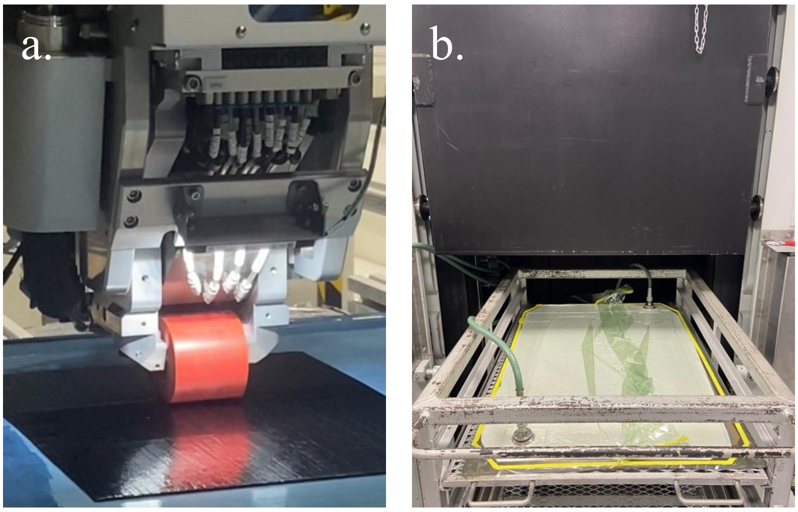

Figure 1. Composite skin manufacturing a. lay-up process with automated fiber placement and b. oven curing after 24 hours of debulking.

2.2.2 Plasma Treatment Procedure

In this study, two groups of composite specimens are manufactured and tested, namely, APA-treated (will be referred as "APA" batch) and non-treated ("NT") specimens. A rotating nozzle (2500 rpm) on an atmospheric plasma system (Openair® FG5001, Plasmatreat Inc.) is used for the execution of APA treatment on the surface of the skin laminates [36]. The thermoset-based composite skin is fixed on the table of the plasma treatment system. The surfaces of thermoset-based UD composite skins are cleaned with isopropyl alcohol (IPA) and air-dried at room temperature. Different APA process parameters listed in Table 1 are applied to obtain optimum APA process parameters for this type of manufactured composite skins. Following the APA procedure, water contact angle values (WCA) for all these parameters are measured three times using deionized water with a Kruss FM40Mk2 drop shape analyzer (DSA). The contact angle is recorded after allowing five seconds for each liquid drop to settle. Since the surface energy of deionized water includes both polar and dispersive components, water contact angle measurements provide a comprehensive estimation of the surface energy of interest [37]. Moreover, the availability of deionized water simplifies the surface energy analysis and saves costs. Following this, the APA parameter set that provides the minimum WCA values is chosen for application prior to the adhesive bonding procedure. Moreover, water contact angle measurement of non-treated (NT) surfaces is conducted right after cleaning with IPA and drying.

Table 1. Set of APA process parameters implemented in this study.

Parameters	**P1**	**P2**	**P3**	**P4**	**P5**	**P6**	**P7**	**P8**	**P9**	**P10**	**P11**	**P12**
Scan speed (mm/sec)	20	20	20	30	30	30	20	20	20	30	30	30
Nozzle Distance (mm)	15	15	15	15	15	15	20	20	20	20	20	20
Scan Number	1	2	3	1	2	3	1	2	3	1	2	3

2.2.3 CFRP Nomex Sandwich Composite Manufacturing

For the manufacturing of sandwich composites, the process begins with cutting of the cured composite laminates, each having a thickness of 1.3 mm, into precise dimensions of 200×200 mm^2 using a KUKA KR-16-2 CF robotic waterjet machine (Figure 2a). Similarly, Nomex honeycomb cores are cut to the same dimensions using a ZÜND G3-L3200 digital cutting machine (Figure 2b). Both the skin laminates and honeycomb cores, once cut to the required dimensions, undergo an overnight drying process in an SLW 115 IG SMART / Pol-Eco-Aparatura oven. The temperature is increased from ambient (22.5°C) to 75°C to remove residual moisture in the material (Figure 2c). Regarding the adhesive film preparation, Scotch-Weld AF 163-2 K adhesive films, initially stored at -18°C, are maintained at a controlled temperature of 4°C to prevent moisture accumulation. After a 30-minute conditioning period at room temperature, these films are precisely cut to the required dimensions using the ZÜND G3-L3200 digital cutting machine (Figure 2d).

Following, optimum APA process parameters, which are previously specified by using the minimum WCA values, are implemented on the carbon fiber-epoxy top and bottom composite skins (Figure 2e). These APA-treated skins are then bonded to the Nomex honeycomb core using 3M/AF 163-2 K structural adhesive, whereas NT laminates are directly bonded without any APA treatment, with the same adhesive. Clamping elements are used on both sides of the sandwich structures to prevent slippage of the uncured adhesive (Figure 2f). Both APA-treated and NT sandwich composites undergo a controlled curing process of the adhesive, in accordance with the specifications outlined in the adhesive manufacturer's data sheet (Figure 2.g). The curing process involves placing the sandwich composites in an LP-MH4H50/MSE manual hot press machine, applying a pressure of 2.5 bar (Figure 2g). The temperature is increased from 20°C to 108°C at a rate of 3°C per minute, followed by a 90-minute dwell period at 108°C. After curing, the sandwich composites are naturally cooled down to an ambient temperature.

2.2.4 Characterization and Mechanical Tests

After manufacturing the sandwich composites, the effect of APA treatment on the bonding performance is evaluated through a series of characterization and mechanical tests. To investigate the chemical groups and the effects of APA treatment on the CFRP sandwich specimens, FTIR spectroscopy (Thermo Scientific Nicolet™ iS50) is employed in transmission mode. The measurement range for the infrared spectroscopy extends from 4000 to 600 cm^{-1}, with a total of 16 scans recorded for each sample at a resolution of 4 cm^{-1}.

Ensuring the integrity of the bond between the core and skin is crucial for maintaining stability and facilitating effective load transfer between the skins and the core. For this purpose, the cross-sections of APA and NT sandwich structures are examined using a Nikon SMZ800N

Stereo Microscope, with a focus on the morphology of the adhesive layer. Subsequently, the performance of the composite structures is assessed through flatwise tensile and Charpy impact tests. Finally, the thermo-mechanical behaviors of the APA and NT specimens are compared using DMA.

In accordance with the guidelines outlined in ASTM C297, flatwise tensile test specimens for each APA and NT groups are subjected to waterjet cutting into the dimensions of 50×50 mm^2 with a uniform thickness of 12.44 mm, resulting in a set of four specimens to each APA and NT sandwich composites. To remove any residual moisture from the specimens cut with a waterjet, an overnight drying process at 75°C is carried out.

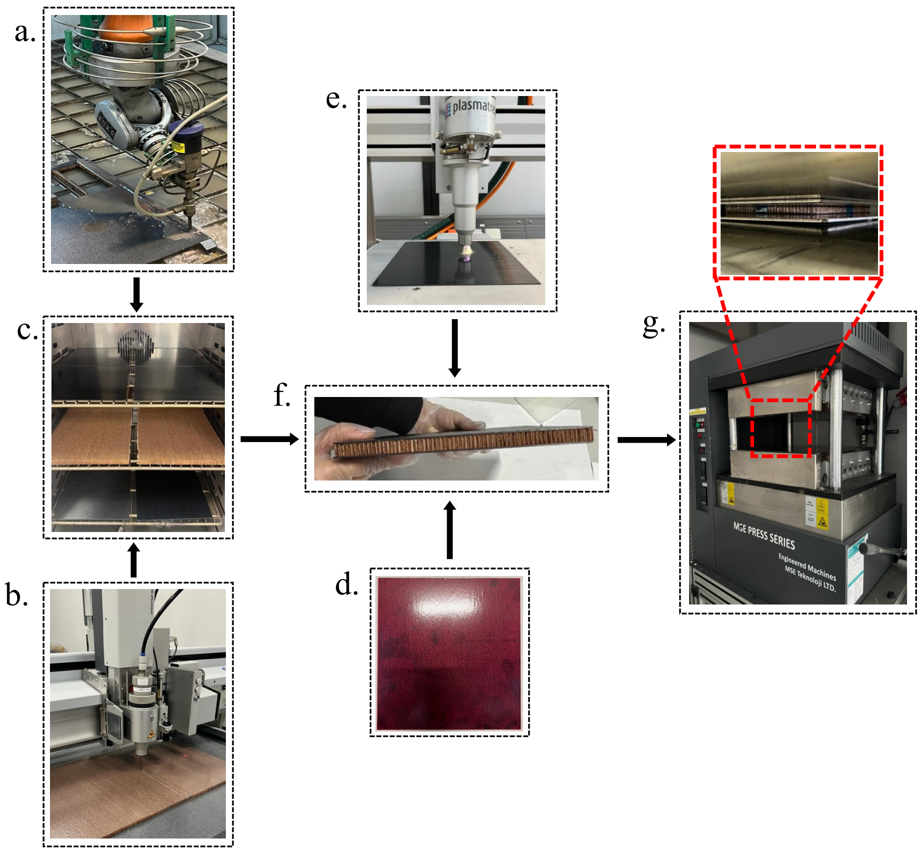

Figure 2. Composite sandwich manufacturing: a. Waterjet cutting of skin laminates, b. Digital cutting of honeycomb, c. Oven drying of skin and honeycomb cores, d. Preparation of 3M/AF 163-2 adhesive film, e. APA treatment of the composite specimens, f. Uncured

sandwich composite fixed to prevent slippage of adhesive layer, g. Curing of adhesively bonded honeycomb structure using hot press process.

The schematics of the flatwise tensile testing of the honeycomb composite structure is presented in Figure 3. The test systems include two aluminum blocks, adhesively bonded to the top and bottom skins of the honeycomb structure, and two stainless steel fixtures through which the load is applied and transferred to the specimen. A critical aspect of the flatwise tensile testing is the proper preparation of the aluminum block surfaces and consistent application of adhesive to ensure the test validity by preventing any failure in the epoxy bonding between the aluminum blocks and the specimen [38]. To enhance the adherence of the specimens to the test fixture, the aluminum alloy blocks are subjected to a cleaning procedure in accordance with ASTM D3933 [39]. This cleaning process involves multiple stages [40]. Initially, the blocks are cleansed using a solvent known as isopropyl alcohol (IPA). Subsequently, an alkaline liquid solution is prepared. This solution is composed of 40 grams of sodium hydroxide (NaOH) with the addition of 2000 milliliters of distilled water. The blocks are immersed in the alkaline solution for an approximate duration of 15 minutes. In the third step, a solution consisting of approximately 10% phosphoric acid is prepared by blending 195 milliliters of phosphoric acid with 3000 milliliters of distilled water. Following this, the aluminum alloy blocks are immersed in the solution, with only one surface being subjected to electrochemical treatment at a voltage of 10 volts for a duration of 25 minutes.

Once the electrochemical treatment is completed, the blocks are removed from the solution and are transferred to a container filled with distilled water at room temperature and held in the container for approximately 15 minutes. As a concluding step, the aluminum blocks undergo a drying process in an oven set at 80°C for 30 minutes.

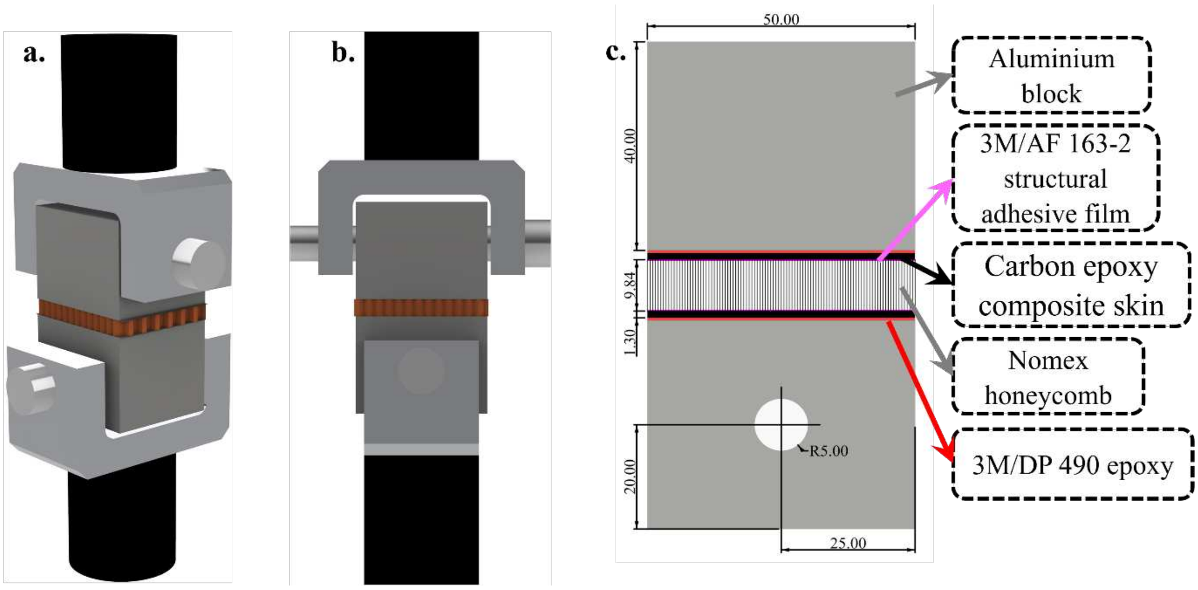

Figure 3. Schematic illustration of flatwise tensile test system: a. Isometric view, b. frontal view, and c. the configuration of the test components and the specimen adhered to the blocks.

The flatwise tensile testing of APA and NT specimens is conducted using an INSTRON model 5982 testing machine, with a maximum load of 100 kN and at a speed rate of 4.00 mm/min (Figure 3). The test is repeated four times for each APA and NT specimen group. The aluminum blocks are bonded to the APA and NT specimens with the 3M Scotch-Weld DP 490 epoxy structural adhesive. After bonding the specimens to the aluminum blocks, the specimens are allowed to cure for 24 hours at room temperature.

The Charpy impact test is essential for evaluating the impact performance and reliability of composite sandwich structures, particularly for assessing durability and core-skin bonding. APA and NT impact test specimens are prepared according to the ASTM D6110 standard. Figure 4 schematically illustrates both the specimen setup (Figure 4.a) and the impact test procedure (Figure 4.b). These specimens are precisely cut to dimensions of 130×15 mm^2 at a thickness of 12.44 mm using the robotic waterjet machine and four test specimens for each APA and NT groups are prepared. Due to the high tensile strength of the carbon fiber composite within the sandwich structure, a 50-joule hammer is employed during testing. The hammer, serving as the testing equipment, is positioned in the fixture to strike the unnotched specimens at the skin region (Figure 4.b). Following the Charpy impact test, to develop a further understanding of the failure modes, microscopic analysis of the fractured impact test specimens is conducted using a Leo Supra 35VP field emission scanning electron microscope (FE-SEM) at an accelerating voltage of 5 kV. For this purpose, the fractured APA and NT specimens are cut to approximately 10 mm in length using an electric saw. To enhance electrical conductivity, a thin layer of Pd/Au is applied onto the SEM samples.

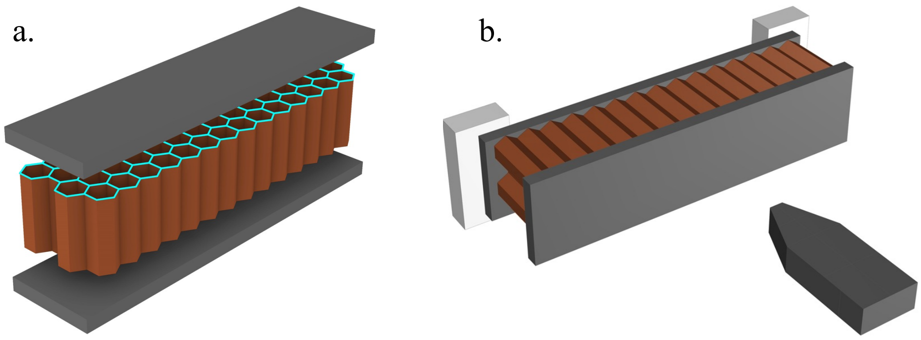

Figure 4. Schematic representation of Charpy impact test: a. components of the sandwich specimen and b. testing configuration.

The effect of APA treatment on the thermomechanical behavior of the composite laminates is further evaluated through DMA. This technique enables the measurement of mechanical properties with respect to time, temperature, and frequency [41]. In this study, three specimens are prepared for both the APA and NT groups, each measuring 55×10 mm² with a thickness of 12.44 mm. Figure 5 provides rendered front, side, and perspective views of the specimen, showing its placement within the experimental setup. This test is conducted using the Mettler Toledo DMA/SDTA861e instrument. The experimental procedure follows dual cantilever mode, as specified in ASTM D7028-07. The temperature range for the experiments is from 25°C to 200°C, with a heating rate of 3°C per minute, and analysis is performed at a frequency of 1 Hz.

a. b.

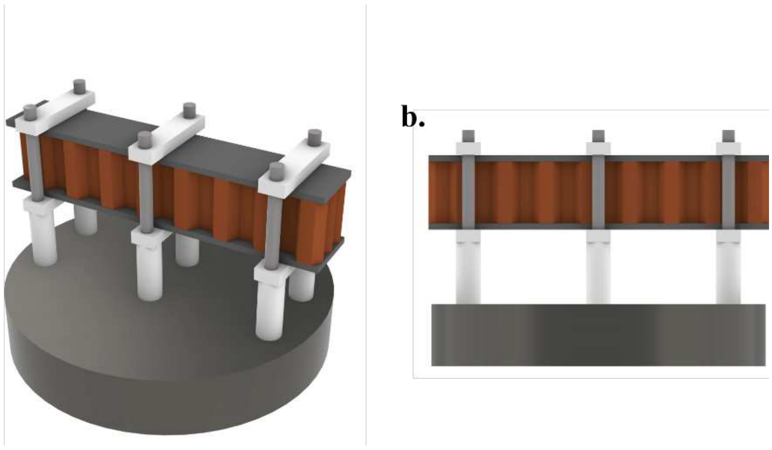

Figure 5. Schematic representation of the DMA specimen and testing in double cantilever mode: a. perspective view and b. front view.

3 Results and Discussion

3.1. Contact Angle Measurement Results

Figure 6a presents the contact angle results for NT and APA sample groups. The data shows a decrease in WCA values following the APA treatment. The effectiveness of APA treatment, namely, the reduction in the WCA values, is closely related to the optimal balance between treatment distance and duration [42]. Conversely, when the scan speed remains constant and the nozzle distance increases, higher WCA values are obtained (P1-7, P2-8, P3-9). Additionally, as the scan number increases across all treatment conditions, higher WCA values are obtained (P1-2-3; P4-5-6; P7-8-9; P10-11-12). This increase is likely due to enhanced interaction between the surface and the plasma nozzle, potentially causing surface damage [21]. Among all parameter sets, P4 (scan speed 30 mm/sec, nozzle distance 15 mm, and scan number 1) is identified as the optimal condition, resulting in the lowest WCA values implying highest wettability [43]. Representative WCA images for NT and APA-treated (P4 parameter set) surfaces are shown in Figure 6b-c, respectively. Based on this analysis, during sandwich panel manufacturing, P4 APA parameter sets are employed for bonding the skin surfaces to the honeycomb cores, as described in Section 2.2.3 (Figure 2e).

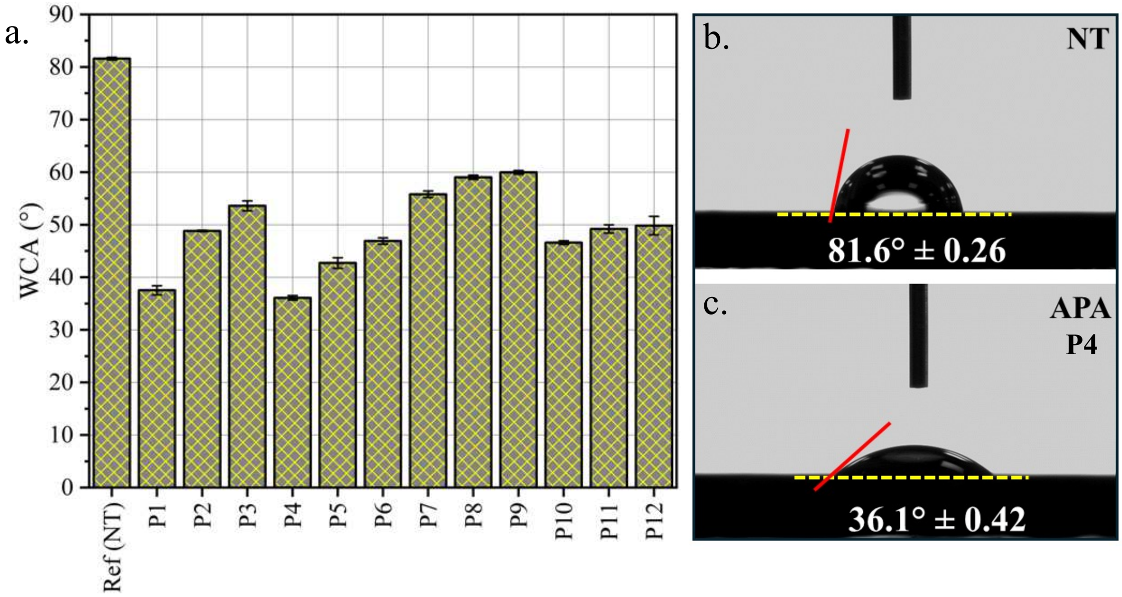

Figure 6. a. WCA values for NT and APA treated specimens; representative WCA images of b. NT and c. APA-treated surfaces.

To examine the effect of APA treatment on the chemical functional groups of the treated surfaces, FTIR analysis is conducted, as the transmittance percentages are presented in Figure S1. The peaks located at 3100–3500 cm^{-1}, 3000–2700 cm^{-1}, and 1650–1800 cm^{-1} correspond to OH, C–H, and C=O bonds, respectively [44,45]. A comparison of the FTIR results for the NT and APA specimens reveals no significant differences in peak positions or the appearance of new peaks, indicating that APA treatment does not damage or disintegrate the treated surfaces. Upon examination of the FTIR results for the NT sandwich composite specimen, two partially overlapping peaks appear around 1724 cm^{-1}, as shown in Figure S1. Following APA treatment, these peaks broaden and completely overlap, resulting in a single peak at 1744 cm^{-1} (Figure S1). Since the peaks in this range (1650–1800 cm^{-1}) correspond to C=O bonds, the APA treatment leads to the formation of new oxygen-related functional groups [46]. This is expected, as the surface absorbs oxygen during APA treatment, leading to the creation of these new functional groups. This finding aligns well with the mechanical results that will be discussed in the following sections.

3.2. Microscopic Analysis of APA Effect on Adhesive Morphology

Microscopy images of APA (Figure 7a) and NT (Figure 7b) sandwich composite specimens, captured at different magnifications using a Nikon SMZ800N Stereo Microscope, reveal distinct differences in adhesive distribution at the skin – core interface cross sections. In the APA specimens, the adhesive exhibits a uniform and homogeneous distribution throughout the skin-core interface, attributed to the enhanced wetting feature thanks to APA process prior to adhesive application, resulting in consistent bonding characteristics, as shown in Figure 7.a. Conversely, the NT sandwich specimens display an irregular adhesion pattern at the interface cross section, with a non-uniform adhesive distribution, as depicted in Figure 7.b.

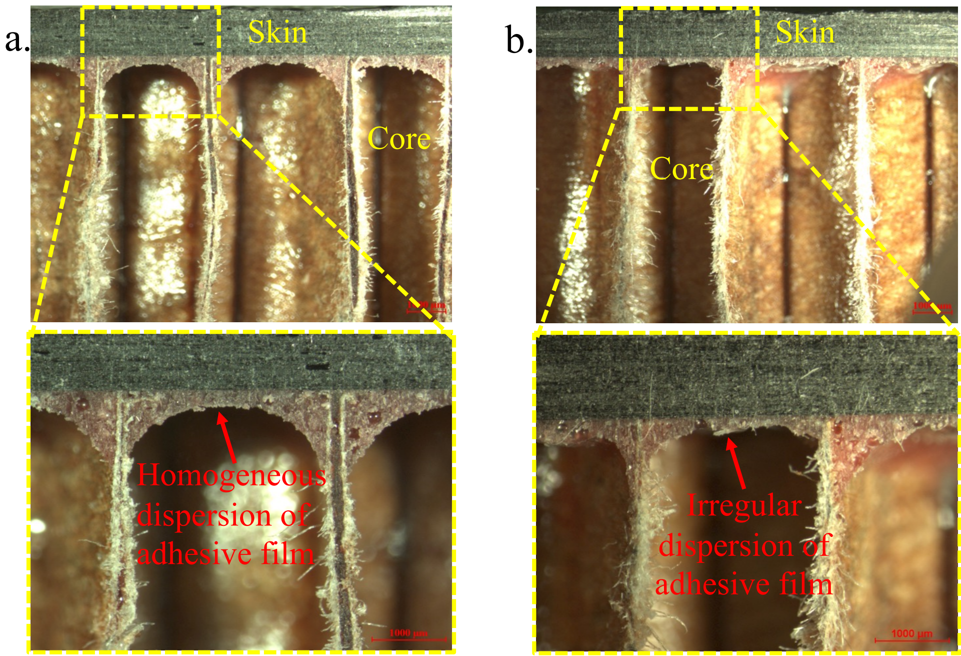

Figure 7. Microscopy images of cross-sections of a. APA-treated and b. NT specimens.

3.3.Flatwise Tensile Test Results

Figure 8 presents the flatwise tensile test results in the form of tensile stress-tensile displacement graph (Figure 8a) and bar plot for the average tensile strength (Figure 8b) for APA and NT specimens. The APA specimens exhibit an average tensile strength value of 2.4 MPa and an average maximum load of 5.7 kN, whereas the NT specimens demonstrate corresponding values of 2.17 MPa and 5.23 kN, respectively. Liu et al. performed flatwise tensile tests on carbon fiber reinforced Nomex honeycomb sandwich composites with a [45/−45]s stacking sequence, noting an average tensile strength of approximately 2.13 MPa [29]. Additionally, Zhu et al. conducted flatwise tensile tests on Nomex core sandwich composites and reported an average tensile strength of 2.18 MPa [31]. Their findings are comparable to the average tensile strength observed for the NT specimens in the current study. Moreover, Choi et al. manufactured sandwich composites made of carbon/epoxy skins with a Nomex honeycomb core using secondary bonding. They carried out flatwise tensile tests and reported a tensile strength of approximately 1.6 MPa [47]. A thorough examination of the test results for APA specimens demonstrates 11% higher tensile strength values than the NT specimens. Furthermore, increased displacement under stress indicates enhanced toughness for the APA specimens.

Moreover, in both APA-treated and NT specimens, it is observed that the fracture predominantly occurs within the core, as shown in Figure 9. Complete failure and separation did not occur instantaneously; rather, cracks took some time to fully propagate through the honeycomb under lower load conditions[11]. The differences in the fracture patterns observed between these specimens can be summarized as follows. The fracture of the APA specimen displays a wavy pattern within the honeycomb (Figure 9a), contrasting with the more linear crack advancement behavior in the NT specimen (Figure 9b). Furthermore, the presence of specific fracture regions near the skin region of the NT specimen indicates lower endurance compared to the APA specimen (see the inset figure for detail). This failure can be ascribed to the relatively lower tensile strength of the core structure when compared to the bond strength in the interfacial region between the core and the skin [48].

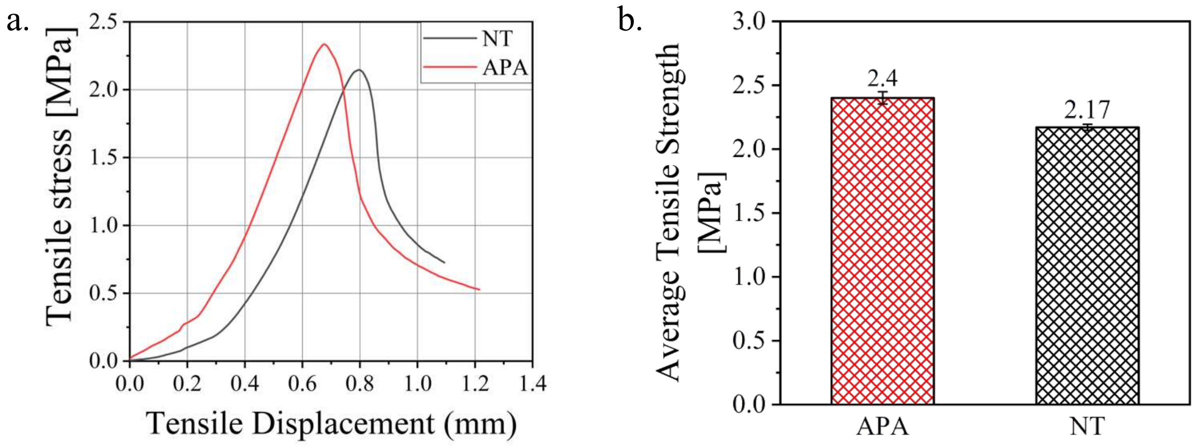

Figure 8. a. Tensile stress-displacement curves of APA-treated and NT sandwich composite specimens and b. Average tensile strength values of APA-treated and NT sandwich composite specimens.

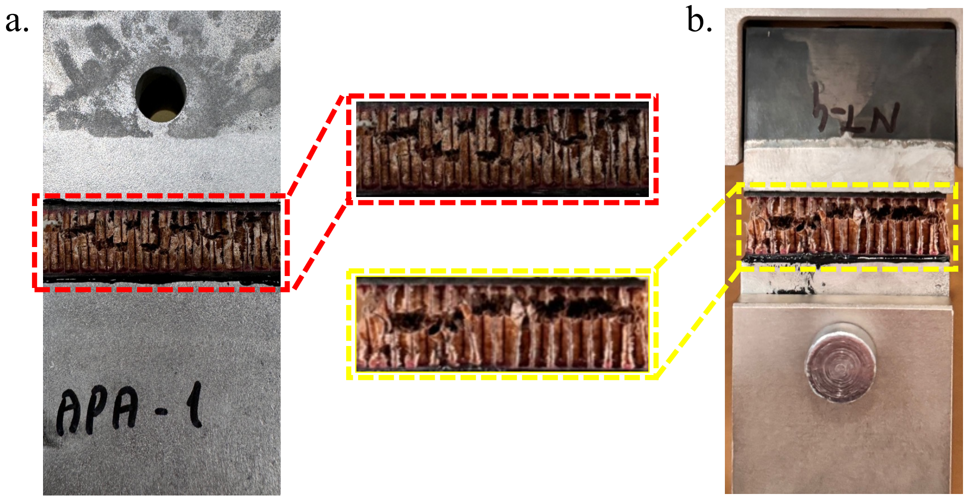

Figure 9. The fractured region of Nomex honeycomb core after flatwise tensile tests: a. APA-treated and b. NT specimens.

3.4.Charpy Impact Test Results

The Charpy impact test is important for assessing toughness under an external load, as it measures the amount of energy absorbed by a material prior failure. This is a critical parameter for evaluating the performance of sandwich composites [49]. When these structures are subjected to Charpy impact testing, the impact energy is concentrated along the thickness of the sandwich structures, leading to fractures originating from the impact point [50]. The Charpy impact test results, presented in Figure 10, show the average impact strength values for four specimens in both APA-treated and non-treated (NT) types. The NT specimens, which have a skin thickness of 1.3 mm, demonstrate an average impact energy of 110.87 kJ/m², aligning well with the results reported in the literature[26]. APA specimens demonstrate 12% higher average impact energy value with 124.66 kJ/m², thereby augmenting the impact absorption capability of the sandwich composite. This improvement can be attributed to the homogeneous dispersion of the adhesive material between the atmospheric plasma activated skin surface and the honeycomb core structure, leading to a mechanical enhancement (Figure 7.a).

Additionally, the Charpy impact test findings are elaborated with SEM imaging of the fractured specimens. The SEM image presented in Figure 11a illustrates the occurrence of fiber fracture within the impacted skin part of the impact specimen treated with APA. This observation implies the presence of favorable adhesion characteristics. The SEM image displayed in Figure 11b reveals a discernible separation within the adhesion zone of the NT sample following the impact. Skin-core debonding occurs because of the bonding strength connection. Thus, the Charpy impact test results provide empirical evidence, confirming the presence of a weak bonding between the skin layers and the core.

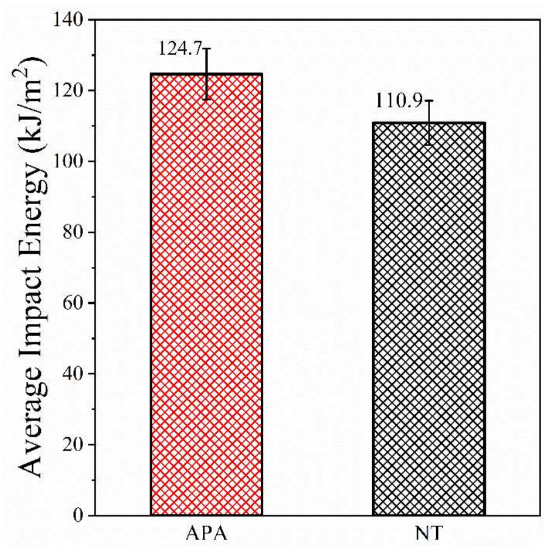

Figure 10. Average impact energy (kJ/m²) values of APA-treated and NT specimens.

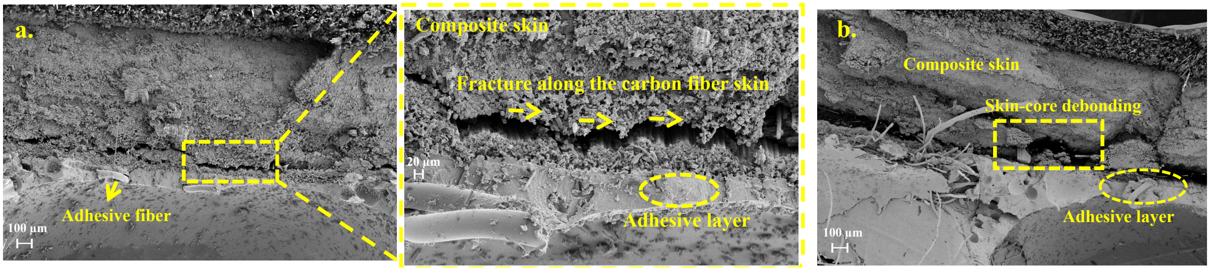

Figure 11. Scanning Electron Microscope (SEM) images for a. APA-treated and b. NT specimens.

3.5. Dynamic Mechanical Analysis (DMA) Results

Within the scope of this study, the storage modulus (E') and loss modulus (E") are measured to assess the material's capability for energy storage and loss, respectively, under applied loads during the experimental procedure. Additionally, the tan (δ), calculated as the ratio of the loss modulus to the storage modulus, has been employed as a quantitative measure to evaluate the energy loss characteristics of the materials under investigation.

Figure 12 illustrates the curves of APA and NT sandwich composite specimens obtained from the DMA test conducted concerning E', E" and tan (δ). It can be seen from Figure 12a that although the storage modulus of APA specimen is higher than that of NT specimen at room temperature, indicating improved interfacial bonding [51], it drops below that of NT specimen shortly after the temperature is increased. The more pronounced decline of the APA specimen suggests a more efficient heat transfer between the skin and the core materials compared to the NT specimen. This efficient heat transfer enables the core of the APA specimen to heat faster than its corresponding in NT specimen. This helps the core in the APA specimen to soften faster which leads to a quicker depreciation in the storage modulus of APA specimen. The reason behind the efficient heat transfer between the skin and the core material in the APA specimen is attributed to the superior bonding facilitated by the APA treatment which agrees well with the mechanical testing results.

Figure 12b presents the loss modulus results for APA and NT sandwich composite specimens. In the NT specimen it can be seen that the two loss modulus peaks corresponding the T_g temperature of the phenolic resin of the core material and the epoxy resin of the skin material overlap [52]. This overlap is less pronounced in the case of APA specimen which supports discussions concerning the storage modulus results. As the heat transfer is more efficient in APA specimen, the core material reaches its T_g temperature faster than the core material in NT specimen. Furthermore, the reduced overlap is also visible in tan (δ) results provided in Figure 12c where the shoulder in the graph of APA specimen is more protruding when compared with the tan (δ) graph of NT specimen. Additionally, the tan (δ) peak of APA specimen is higher than that of NT specimen because of the lower storage modulus of APA specimen at elevated temperatures.

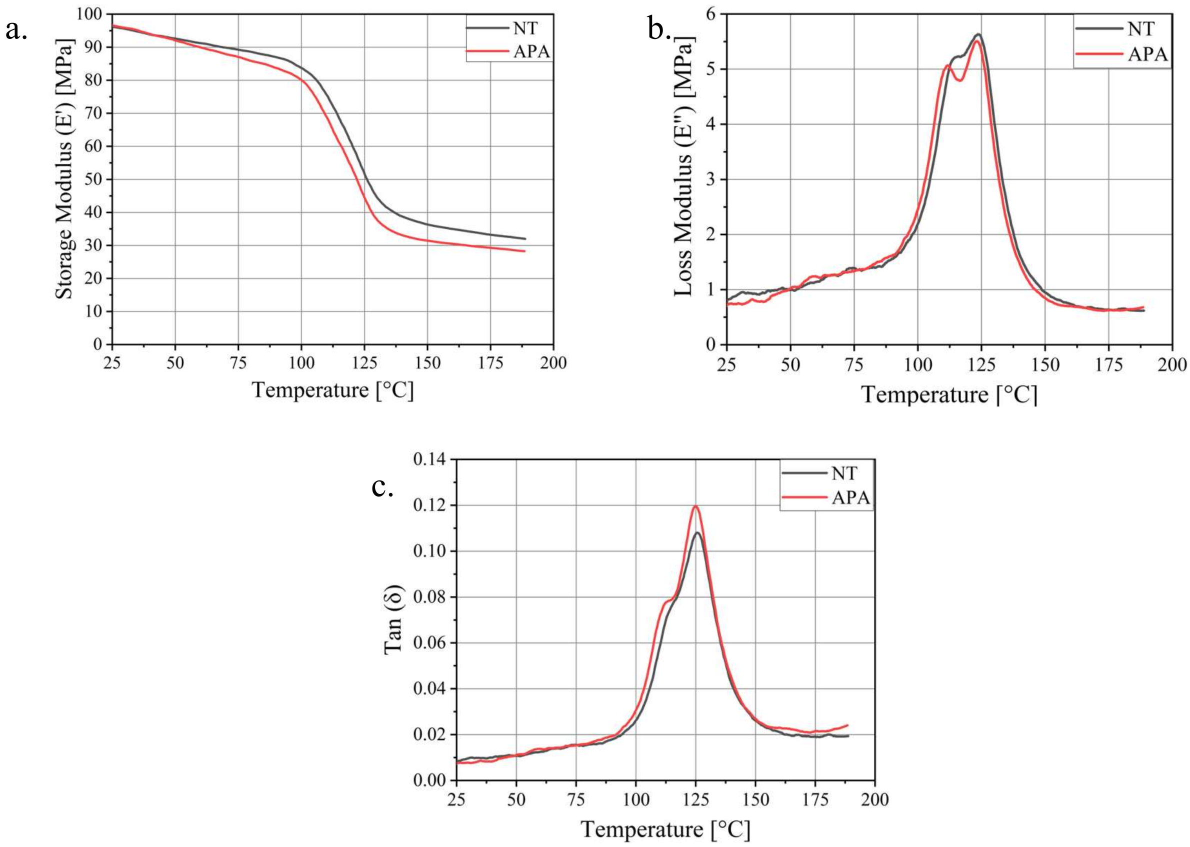

Figure 12. DMA curves of APA -treated and NT specimens for a. Storage Modulus (E'), b. Loss Modulus (E") and c. Tan (δ).

4. Conclusion

This study demonstrates the significant benefits of Atmospheric Plasma Activation (APA) treatment on composite sandwich panels, focusing on improving adhesive performance and mechanical properties. APA treatment effectively enhanced surface wettability, leading to a more uniform adhesive distribution across the honeycomb core of the sandwich structures. This uniformity translated into notable improvements in tensile strength and impact resistance. Specifically, APA-treated specimens exhibited an 11% increase in tensile strength and a 12% improvement in impact strength compared to non-treated counterparts, highlighting the treatment's effectiveness in enhancing panel durability.

The flatwise tensile tests revealed that APA-treated panels not only achieved higher tensile strength but also displayed better toughness, with fracture patterns indicating more robust adhesion. The Charpy impact tests further corroborated these findings, showing that APA-treated panels absorbed more impact energy, reflecting enhanced mechanical resilience. Moreover, the SEM analysis provided additional validation of the enhanced bonding quality in APA-treated panels, while non-treated panels exhibited signs of weaker adhesion. Dynamic Mechanical Analysis (DMA) confirmed that APA treatment improves the energy absorption characteristics of the panels, with APA-treated specimens demonstrating superior toughness.

In summary, APA treatment emerges as a highly effective technique for enhancing the performance of composite sandwich panels, offering improved adhesive bonding, greater mechanical strength, and better impact resistance. This approach holds significant promise for applications requiring high-performance composite materials, contributing to advancements in the field of material science and engineering.

Acknowledgement

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Data Availability

Data will be made available on request.

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Enhancing Adhesive Bonding and Mechanical Properties of Composite Sandwich Panels through Atmospheric Plasma Activation

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S1. Fourier Transform Infrared Spectroscopy (FTIR) tests

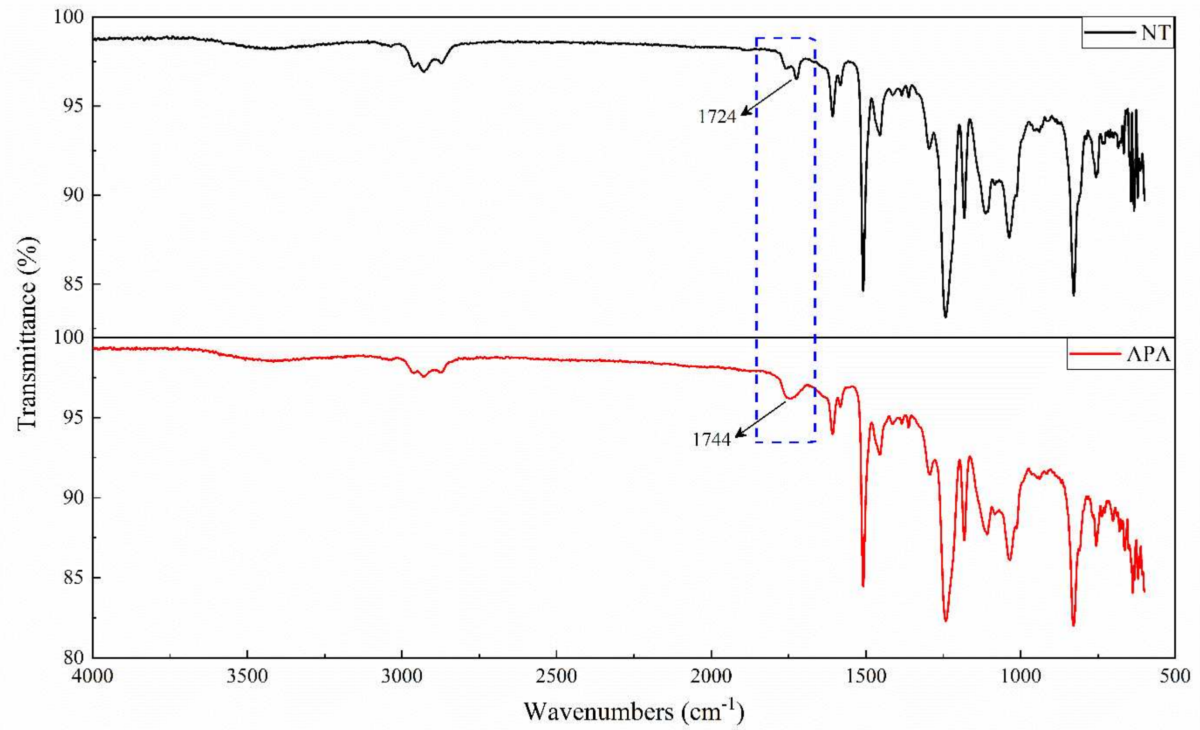

Figure S1. Fourier-transform infrared spectroscopy spectrums of before and after the atmospheric plasma activation (APA) of composite skins.